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## PRODUCTION OF ALUMINUM NITRIDE BASE PLATE AND SINTERING VESSEL

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### Abstract

**PURPOSE:** To improve thermal conductivity and surface roughness by accommodating AlN base plate compacts in a cylindrical vessel made from an AlN sintered compact, putting covers made from a BN sintered compact on the vessel and sintering the base plate compacts in a non-oxidative atmosphere.

**CONSTITUTION:** In a sintering vessel 6 composed of a cylindrical vessel body 6a made from an AlN sintered compact and covers 6b and 6b made from a BN sintered compact, base plate compacts 4 composed mainly of an AlN powder having 0.05 to 5μm average particle diameter and exhibiting ≤3wt.% impurity oxygen content are put through placing powder 7 composed of an AlN sintered powder having 50 to 200μm average particle diameter in layers. The sintering vessel 6 is accommodated in an oven 1 and sintering is executed in a non-oxidative atmosphere at 1600 to 2000 deg.C for 2 to 10hr.

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[Title of the Invention]

PRODUCING METHOD OF ALUMINUM NITRIDE SUBSTRATE, AND  
FIRING CONTAINER

[Abstract]

[Object]: To improve the durability of a firing container (box) used at firing in the case of producing a large-size aluminum nitride substrate, and produce an aluminum nitride substrate having excellent thermal conductivity and surface roughness in quantity at low cost.

[Constitution]: A producing method comprises sintering a substrate formed body having an aluminum nitride as a main component in a state where the substrate formed body is put in a firing container which is formed of a cylindrical container main body made of an aluminum nitride sintered body and a lid, made of a boron nitride sintered body, for covering the container main body, under non-oxidizable atmosphere.

[Claims]

1. A producing method of an aluminum nitride substrate, comprising: sintering a substrate formed body having an aluminum nitride as a main component in a state where the substrate formed body is put in a firing container which comprises a cylindrical container main body made of an aluminum nitride sintered body and a lid, made of a boron nitride sintered body, for covering the container main body, under non-oxidizable

atmosphere.

2. The producing method of an aluminum nitride substrate according to claim 1, comprising: putting a plurality of substrate formed bodies in the firing container; and interposing an aluminum nitride sintered powder, as a spread powder, between the adjacent substrate formed bodies to arrange the respective substrate formed bodies in a lamination.

3. The producing method of an aluminum nitride substrate according to claim 2, comprising setting the sintering powder as a spread powder so as to have an average particle diameter in a range of 50 to 200  $\mu\text{m}$ .

4. A firing container comprising: a cylindrical container main body made of an aluminum nitride sintered body; and a lid, made of a boron nitride sintered body, for covering the container main body.

[Detailed Description of the Invention]

[0001]

[Field of the Invention]

The present invention relates to a producing method of an aluminum nitride substrate, and a firing container to be used in the producing method and, more particularly, to a producing method of an aluminum nitride substrate capable of improving the durability of a firing container (box) used at firing in the case of producing a large-size aluminum nitride substrate, and producing an aluminum nitride substrate having

excellent thermal conductivity and surface roughness in quantity at low cost, and a firing container.

[0002]

[Prior Art]

Ceramic sintered bodies excellent in a variety of properties such as strength, heat resistance, corrosion resistance, wear resistance, light weight property as compared with those of conventional metal materials have been widely used as mechanical parts, structural materials and ornamental materials, such as semiconductors, electronic device materials, engine parts and materials for a high-speed cutting tool, nozzles, bearings, for use under severe temperature, stress and wearing conditions under which the conventional metal materials could not be used.

[0003]

Especially, an aluminum nitride (AlN) sintered body is an insulator having a thermal conductivity about 20 times as high as that of an  $\text{Al}_2\text{O}_3$  sintered body and has a heat expansion coefficient close to that of silicon (Si) forming a semiconductor element, so that it has been used in a more widened range of applications, as a heat releasing plate and a substrate of a highly integrated semiconductor device, a large-size chip, and a high power module constituent.

[0004]

Conventionally, the above-mentioned aluminum nitride substrate has generally been produced in quantity by the following production method. That is, a raw material mixture is produced by adding a sintering aid such as  $\text{Y}_2\text{O}_3$ ,  $\text{CaO}$  and the like, an organic binder and, based on the necessity, a

variety of additives, solvents, and dispersants to an aluminum nitride powder and the obtained raw material mixture is formed into, for example, a sheet-shape by a doctor blade method, or a thick plate-like or large-size formed body by press forming the mixture. In the case of producing a product with a complicated shape, a molded body is produced by a slip casting method.

[0005]

A standard size of a ceramic substrate in the main stream is generally 2 inch-square (2" × 2") and, based on the applications, those of 3" × 3" and 4" × 4" may be used and further large size substrates with 6" × 6" or larger tend to be used. The forming process of a substrate formed body to produce such a large substrate is easy irrespective of the size and a doctor blade method or a press forming method can be employed therefor.

[0006]

A formed body obtained accordingly is subjected to a degreasing treatment by heating it in air or nitrogen gas atmosphere to degrease and remove carbon, hydrogen, or the components used as an organic binder from the formed body. The degreased formed body is heated at a high temperature in nitrogen gas atmosphere and densified and sintered to produce an aluminum nitride substrate.

[0007]

The above-mentioned sintering process, as illustrated in Fig. 2, is generally carried out by setting a box-like firing container (a box for firing) 3 on a hearth 2 of a firing furnace 1 lined with a refractory such as graphite or the like, putting one or a plurality of degreased substrate formed bodies 4 in

the firing container 3 and in such a state, heating at 1600 to 2000°C.

[0008]

The above-mentioned firing container 3 and hearth 2 for putting and holding the substrate formed bodies 4 are made of an aluminum nitride (AlN) sintered body, which is the same material as that of the formed bodies, or a boron nitride (BN) sintered body in order to prevent deterioration of the characteristics of sintered bodies due to the reaction with the formed bodies at the time of high temperature sintering. The above-mentioned firing container 3 comprises a cylindrical or polygonal tub-shaped container main body 3a, and lids 3b, 3b to cover an upper end opening and a lower end opening, respectively, to seal them. In the case a plurality of substrate formed bodies 4 are arranged in layers in the firing container 3 and simultaneously fired, a BN powder 5 with a particle size of about 1  $\mu\text{m}$  as a spread powder (a release agent) is interposed between adjacent substrate formed bodies 4, 4.

[0009]

As described above, since the substrate formed bodies 4 are put in a firing container which does not react on the substrate formed bodies 4, the respective substrate formed bodies 4 are evenly heated as a whole to obtain even sintered bodies with little unevenness. Further, impurities such as carbon contained in the refractory of the firing furnace 1 does not affect directly the formed bodies 4 at the time of firing and uneven coloration and deformation in the sintered bodies owing to the impurities can be thus prevented and so can decrease of the thermal conductivity of each sintered body. Further, since the BN powder 5 as a spread powder is interposed

among a plurality of substrate formed bodies 4, 4, adjacent formed bodies have no risk to be fused each other to result in production of aluminum nitride substrates with excellent surface roughness.

[0010]

On the other hand, in the case where substrate formed bodies 4 are arranged directly on a firing furnace 1 without using the foregoing firing container 3, the surfaces of the sintered bodies are considerably damaged by an excess amount of carbon vapor released from the refractory of the firing furnace 1 and the entire sintered bodies expand their deformation degree to result in sharp decrease of the production yield.

[0011]

In the above-mentioned production method, in the case where an ultrafine raw material powder with an average particle diameter of about  $0.3\ \mu\text{m}$  or less is used as a raw material AlN powder, a rather dense sintered body can be obtained even from an AlN powder alone. However, at the time of sintering, an impurity of such as a large quantity of oxygen or the like adhering to the surface or the like of a raw material powder forms a solid solution in the AlN crystal lattice or a compounded oxide of such as Al-O-N compound which inhibits transmission of the lattice oscillation is produced to result in low thermal conductivity of an AlN sintered body in the case of using no sintering aid.

[0012]

Meanwhile, in the case of using an AlN powder with an average particle diameter of  $0.5\ \mu\text{m}$  or more as a raw material powder, since the sintering property of the raw material powder alone is insufficient, it is

difficult to obtain a dense sintered body with no additive addition except by a hot pressing method to result in low productivity. Accordingly, in order to efficiently produce a sintered body by a normal pressure sintering method, as sintering aids, rare earth oxides such as yttrium oxide ( $Y_2O_3$ ) and alkaline earth metal oxides such as calcium oxide are generally added to densify a sintered body and prevent oxygen, an impurity, contained in the AlN raw material powder from forming a solid solution in the AlN crystal particles.

[0013]

It is supposed that these sintering aids form liquid phases by reaction with impurity oxygen contained in the AlN raw material powder to densify the sintered body and immobilize the impurity oxygen in grain boundary phases to achieve high thermal conductivity.

[0014]

[Problems to be Solved by the Invention]

However, in the case of producing an especially large-size AlN substrate according to the above-mentioned conventional producing method, a firing container to be employed has to be enlarged and the production cost of an AlN substrate tends to rise attributed to deterioration of the durability of the firing container. That is, while a firing container made of a BN sintered body has an advantage that it is excellent in high temperature strength as compared with a firing container made of AlN and is scarcely deteriorated by reactions, it has a disadvantage in that its raw material is extremely costly and has another disadvantage in that it is easy to be cracked or fractured to result in cost up of the production cost of a substrate.



[0015]

On the other hand, while a firing container made of an AlN sintered body is economical as compared with a firing container made of BN and has an advantage that the structural strength is sufficiently high in the case of normal handling, its strength is significantly deteriorated at a high temperature and it is easy to be broken in sintering pattern of normal temperature raising ratio to make repeat use impossible.

[0016]

As described above, both of firing containers made of an AlN sintered body and a BN sintered body have such advantageous points and disadvantageous points and especially they both become easy to be broken by thermal impact along with the tendency of enlargement in size and therefore their use and applications have been limited in a restricted narrow range of conditions.

[0017]

Further, in the case of firing a plurality of AlN bodies by a conventional production method, a fine BN powder with a particle size of 1  $\mu\text{m}$  is put between adjacent formed bodies to prevent fusion of the formed bodies each other, however the BN powder adhering to and remaining in the AlN substrate surface on completion of the sintering becomes much to result in a problem of roughening the surface. Therefore, it is required to finish the AlN substrate surface by honing and polishing in order to obtain a prescribed smooth surface and that makes the AlN substrate production process complicated.

[0018]

The present invention has been achieved to solve the above-mentioned problems, and it is an object of the present invention to provide a producing method of an aluminum nitride substrate, and a firing container to be used in the producing method and, more particularly, to provide a producing method of an aluminum nitride substrate capable of improving the durability of a firing container (box) used at firing in the case of producing a large-size aluminum nitride substrate, and producing an aluminum nitride substrate having excellent thermal conductivity and surface roughness in quantity at low cost, and a firing container.

[0019]

[Means for Solving the Problems]

In order to achieve the above-mentioned object, the present inventors have repeatedly made investigations into the relations of materials and durability of firing containers with sintering patterns and the effects of types and particle sizes of spread powders on the thermal conductivity and the surface roughness of an AlN substrate.

[0020]

As a result, it has been found that while a firing container comprising a container main body and lids all made of a BN sintered body has excellent thermal impact resistance in almost all of sintering patterns, lids made of an AlN sintered body are inferior in the thermal impact resistance and easy to be broken unless the temperature rise rate is set to be lower by about 50% than a normal rate. However, a container main body made of an AlN sintered body is confirmed to have excellent thermal impact resistance even if it is repeatedly used at normal temperature rise

rate.

[0021]

On the other hand, when the effect on the thermal conductivity, which is a main characteristic property of an AlN substrate to be a product, is investigated, even though it is partially, the thermal conductivity of an AlN substrate sintered by using a firing container made of AlN is found improved significantly more by 10 to 20% than that sintered by using a firing container entirely made of BN.

[0022]

Further, it has been also found that, in the case of simultaneously firing a plurality of substrate formed bodies, if sintering is carried out while a sintered powder with a prescribed particle size obtained by granulating and sintering an aluminum nitride powder as a spread powder being interposed between adjacent substrate formed bodies, fusion between the adjacent substrate formed bodies can be solved and is remarkably suppressed as compared with that in the case of using a BN powder and AlN substrates with desirable surface roughness can be thus obtained.

[0023]

The present invention has been completed on the basis of the above-mentioned findings. More specifically, a producing method of an aluminum nitride substrate according to the present invention comprises: sintering a substrate formed body having an aluminum nitride as a main component in a state where the substrate formed body is put in a firing container which comprises a cylindrical container main body made of an aluminum nitride sintered body and a lid, made of a boron nitride sintered

body, for covering the container main body, under non-oxidizable atmosphere.

[0024]

Further, it is preferable to put a plurality of substrate formed bodies in the firing container and interpose an aluminum nitride sintered powder, as a spread powder, between the adjacent substrate formed bodies to arrange the respective substrate formed bodies in a lamination.

[0025]

Furthermore, it is preferable to set the sintering powder as a spread powder so as to have an average particle diameter in a range of 50 to 200  $\mu\text{m}$ .

[0026]

Also, an aluminum nitride substrate production method performed by modifying the foregoing method in the foregoing manner.

[0027]

A powder with an average particle diameter of about 0.05 to 5  $\mu\text{m}$ , preferably 3  $\mu\text{m}$  or less and a impurity oxygen content suppressed to 3% by weight or less in consideration of sintering properties and thermal conductivity is used as the aluminum nitride (AlN) powder as a main component of a substrate formed body to be used for a method of the present invention.

[0028]

As a sintering aid, oxides and nitrides of rare earth elements (Y, Sc, Ce, Dy and the like), oxides of alkaline earth metals (Ca), and substances to become these compounds by sintering process may be used and especially,

yttrium oxide ( $Y_2O_3$ ) and calcium oxide (CaO) are preferable. The addition amount of a sintering aid is adjusted in a range from 0.5 to 10% by weight. If the addition amount is less than 0.5% by weight, the effect to improve the sintering properties is insufficient so that a sintered body cannot be densified to result in low strength of a sintered body or oxygen may form a solid solution in AlN crystal to result in production failure of a sintered body with a high thermal conductivity. On the other hand, if the addition amount exceeds, exceeding 10% by weight, grain boundary phases remain~~x~~ in a sintered body or the volume of the grain boundary phases to be removed by thermal treatment is so high that voids remain in the sintered body to result in increase of shrinkage ratio and easiness of deformation.

[0029]

As a constitutional material of a firing container to be used at the time of sintering, at least one of an aluminum nitride (AlN) powder and a boron nitride (BN) powder that do not react on an AlN formed body is prepared. Especially, in a firing container composed of a cylindrical container main body and flat plate-shaped lids to cover the main body, the lids to which high thermal stress is applied at the time of firing is made of a BN sintered body excellent in thermal impact resistance and the cylindrical container main body having a high structural strength is made of an economical AlN sintered body, so that the production cost of the firing container can be remarkably down without deteriorating the durability of the firing container as a whole.

[0030]

As the foregoing firing container, in order to suppress the effect on a

formed body as much as possible, it is preferable to produce the firing container from a material with a high purity that does not react on the formed body and scarcely contains impurities as described above and it can be produced by, for example, the following production method. That is, a firing container is produced by forming a raw material powder mixture obtained by mixing an aluminum nitride powder and an organic binder to be a container original body by a variety of forming methods such as press forming, slip casting, injection molding and the like and after degreasing treatment, sintering the container original body by heating at 1800 to 2000°C for several ten hours in reducing atmosphere of nitrogen gas or the like. Since BN containers are commercialized, large size containers with a size of about  $\phi 400$  and various forms can be produced.

[0031]

On the other hand, as a forming method of the a raw material mixture of a product composing an aluminum nitride substrate, a widely used die pressing method, an isostatic pressing method, or a sheet forming method such as a doctor blade method can be employed. Successively to the forming process, substrate formed bodies are heated at 400 to 500°C in a non-oxidizing atmosphere, for example, in nitrogen gas atmosphere to sufficiently remove the previously added organic binder.

[0032]

Next, the substrate formed bodies subjected to the degreasing treatment are laminated in multiple laminations while an AlN sintered powder 7 being interposed among them in a firing container 6 formed of a container main body 6a made of an aluminum nitride sintered body and lids

6b, 6b made of boron nitride sintered bodies and covering the upper and lower faces of the container main body 6a as illustrated in Fig. 1.

[0033]

Here, the foregoing sintering powder 7 works as a spread powder (a release agent) to prevent fusion of adjacent substrate formed bodies 4 at a time of sintering at a high temperature. As a spread powder to be used for the method of the present invention, a coarse AlN sintered powder whose average particle diameter is set within a range from 50 to 200  $\mu\text{m}$  is used. If the powder is as fine as the average particle diameter less than 50  $\mu\text{m}$ , the atmospheric gas cannot be sufficiently circulated among the substrate formed bodies 4 and further degassing is inhibited so that the composition of the substrate sintered bodies easily become uneven and the amounts of the sintered powder adhering to and remaining in the substrate surfaces increase to required many steps of finishing process of the surfaces. On the other hand, also if the powder is as coarse as the average particle diameter exceeding 200  $\mu\text{m}$ , the substrate surfaces easily become uneven. The foregoing sintered powder may be produced by degreasing and sintering a granulated powder of a commercialized aluminum nitride raw material powder.

[0034]

The sintering process of substrate formed bodies housed in the foregoing firing container is carried out by heating the entire body of the firing container 6 containing the substrate formed bodies 4 at 1600 to 2000°C for 2 to 10 hours in non-oxidizing atmosphere of such as nitrogen gas. The sintering may be carried out in nitrogen gas or nitrogen

gas-containing reductive atmosphere. As the reductive gas,  $H_2$  gas or CO gas may be used. In addition, the sintering may be carried out also in vacuum (containing a slight reducing atmospheric gas) and in atmosphere including reduced pressure, increased pressure and normal pressure. If the firing is carried out at a sintering temperature lower than  $1600^{\circ}C$ , dense sintered bodies are hard to be obtained although they depend on the particle size and the oxygen content of the raw material powder.

[0035]

[Advantages]

According to a producing method of an aluminum nitride substrate and a firing container according to the above-mentioned constitution, since the lids of the firing container to which high thermal stress is applied at the time of firing are made of a BN sintered body excellent in thermal impact resistance, meanwhile the cylindrical container main body having a structural high strength is made of an economical aluminum nitride sintered body, so that the production cost of the firing container can be remarkably down without deteriorating the durability of the firing container as a whole and consequently the production cost of an AlN substrate can be lowered.

[0036]

Further, use of an AlN sintered body for partially forming the firing container makes it possible to produce atmosphere easy to reduce  $Al_2O_3$  in the firing container at the time of firing to decrease the amount of  $Al_2O_3$  contained in the formed bodies and accordingly increase the thermal conductivity of the resulting substrate sintered bodies.



[0037]

Further, in the case of simultaneously firing a plurality of substrate formed bodies, since an aluminum nitride powder with a prescribed particle size is interposed as a spread powder between adjacent substrate formed bodies, the amount of the powder adhering to and remaining in the substrate surfaces is small as compared with that in the case of using a conventional fine BN powder as a spread powder. As a result, the roughness of the substrate surfaces is remarkably improved and the number of steps of the finishing process after sintering can be lessened.

[0038]

[Examples]

Next, with reference to the following Examples, the effects of a producing method of an aluminum nitride substrate and a firing container according to the present invention will be described more specifically.

[0039]

Examples 1 to 5

A slurry was prepared by adding 3% by weight of  $Y_2O_3$  (yttrium oxide) as a sintering aid and further a solvent and an organic binder to an aluminum nitride powder with an average particle diameter of 1.5  $\mu m$  and containing 1.0% by weight of oxygen as an impurity and mixing the resulting mixture in wet state for 24 hours. Next, the slurry was formed by a doctor blade method to obtain a sheet-shaped formed body with a thickness of 0.8 mm and the sheet-shaped formed body was punched to obtain a large number of substrate formed bodies with a regular square shape of 200 mm each side and the respective formed bodies were subjected

to degreasing treatment by heating at 375°C for 2 hours in air.

[0040]

On the other hand, an AlN powder with an average particle diameter of 1.5  $\mu\text{m}$  was mixed with 5% of a binder and granulated by a spray drying method and the raw material mixture was press-molded to produce original bodies of a firing container. As the original bodies of a firing container, a container main body 6a with an inner diameter of 300 mm, a length (a height) of 30 mm in the axial direction, and a thickness of 6 mm and lids 6b with a diameter of 312 mm and a thickness of 6 mm to cover the main body were produced. The respective original bodies of a firing container were put in a container made of AlN and fired at 1900°C for 6 hours in N<sub>2</sub> atmosphere after being subjected to degreasing treatment by heating at 400°C for 2 hours. Accordingly, firing containers 3 and 6 for producing sintered bodies of Examples 1 to 5 and Comparative Examples 1 to 3 were produced. In addition, the container parts 3a and 3b with same shapes as those of 6a and 6b were purchased.

[0041]

An AlN powder with a high purity and an average particle diameter of 1.5  $\mu\text{m}$  was granulated to have a size of 40 to 200  $\mu\text{m}$ , fired at 1900°C for 4 hours in nitrogen atmosphere, successively cooled, and sieved to obtain an AlN sintered powder as a spread powder.

[0042]

Next, eight substrate formed bodies subjected to degreasing treatment in the foregoing process were arranged collectively in multiple laminations in the firing container 6 as illustrated in Fig. 1 and the firing

container 6 was put in a firing furnace in which N<sub>2</sub> gas was enclosed. For sintering treatment of each batch, a spread powder having an average particle diameter shown in the left column of Table 1 was interposed between adjacent substrate formed bodies. Densifying sintering was carried out by raising the temperature of the inside of the firing furnace 1 to 1850°C and keeping at the temperature for 4 hr to obtain eight AlN substrate sintered bodies with a size of 6" × 6" and a thickness of 0.65 mm according to each Example 1 to 5.

[0043]

#### Comparative Example 1

On the other hand, as illustrated in Fig. 2, AlN substrate sintered bodies according to Comparative Example 1 having the same size were prepared by sintering treatment under the same conditions as those of Example 1, except that the firing container 3 including the container main body 3a that was all made of a BN sintered body was used.

[0044]

#### Comparative Example 2

Further, as illustrated in Fig. 2, AlN substrate sintered bodies according to Comparative Example 2 having the same size were prepared by sintering treatment under the same conditions as those of Example 2, except that the firing container 3 including the container main body 3a that was all made of a BN sintered body was used.

[0045]

#### Comparative Example 3

Furthermore, AlN substrate sintered bodies according to

Comparative Example 3 having the same size were prepared by sintering treatment under the same conditions as those of Example 2, except that the firing container of which all of the container main body and covers were made of an AlN sintered body was used.

[0046]

In order to evaluate the characteristics of the obtained respective AlN substrate sintered bodies of Examples 1 to 5 and Comparative Examples 1 to 3, their thermal conductivities were measured and the arithmetical average roughness Ra (JIS B0601) of the surface of each substrate was measured to obtain the results shown in the right column of Table 1.

[0047]

Further, the durability and the production cost of the respective firing containers that significantly affect the production cost of an AlN substrate were calculated and also shown in Table 1. The durability of each firing container was shown as a relative value of the number of repeatedly usable times until the container main body or the lids were cracked or broken in repeated firing processes to 100, a standardized number, calculated by assuming the number of usable times of the firing container entirely made of a BN sintered body shown in Comparative Example 1 to be 100. The production cost of each firing container was shown as a relative value of the total of the raw material cost and the processing cost covering raw material purchasing, forming, sintering and processing costs to 100, a standardized cost, calculated similarly to the cost in the case of Comparative Example 1.

[0048]

[Table 1]

Sample No.	Material of firing container		Spread powder		AlN substrate property		Firing container	
	Container main body	Lid	Type	Average particle diameter ( $\mu\text{m}$ )	Thermal conductivity ( $\text{W/m}\cdot\text{K}$ )	Surface roughness $\text{Ra}(\mu\text{m})$	Durability	Production cost
Example 1	AlN	BN	AlN sintering powder	50	221	0.2	95	45
Example 2	AlN	BN	BN powder	1	218	0.5	95	50
Example 3	AlN	BN	AlN sintering powder	100	225	0.2	95	45
Example 4	AlN	BN	AlN sintering powder	150	231	0.2	95	45
Example 5	AlN	BN	AlN sintering powder	200	226	0.2	95	45
Comparative Example 1	BN	BN	AlN sintering powder	50	194	0.5	100	100
Comparative Example 2	BN	BN	BN powder	1	201	0.2	100	100
Comparative Example 3	AlN	AlN	BN powder	1	198	0.3	25	30

[0049]

As being made clear from the results shown in Table 1, with respect to AlN substrate sintered bodies according to Examples 1 to 5 obtained by sintering using the firing container 6 formed of the container main body 6a made of the AlN sintered body and lids 6b made of a BN sintered body, the thermal conductivities were increased by about 10% as compared with those of the AlN substrate sintered bodies of Comparative Examples 1 to 3 produced by the firing containers entirely made of a BN sintered body or an AlN sintered body and thus sintered bodies with a high heat releasing capability were obtained.

[0050]

Further, since an AlN sintered powder having a coarse particle size as compared with that of a conventional powder was used as a spread powder, the amount of the spread powder adhering to and remaining in the substrate surfaces was considerably decreased to result in improvement of the surface roughness of the substrates.

[0051]

In addition, while the lids 6b to which high thermal stress was applied at the time of firing were made of a BN sintered body excellent in thermal impact resistance, the cylindrical container main body 6a having high structural strength was made of an economical AlN sintered body, so that the production cost of the firing container 6 could be remarkably lowered without deterioration of the durability of the firing container as a whole to lead to production cost down of the AlN substrates themselves.

[0052]

[Effects of the Invention]

As described above, in a producing method of an aluminum nitride substrate and a firing container according to the present invention, while lids to which high thermal stress is applied at the time of firing are made of a BN sintered body excellent in thermal impact resistance, a cylindrical container main body having high structural strength is made of an economical AlN sintered body, so that the production cost of the firing container can be remarkably lowered without deterioration of the durability of the firing container.

[0053]

Further, a firing container is partially made of an AlN sintered body, so that atmosphere easy to reduce  $\text{Al}_2\text{O}_3$  is produced in the inside of the firing container and the amount of  $\text{Al}_2\text{O}_3$  contained in a formed body is decreased to result in increase of the thermal conductivity of an obtained substrate sintered body.

[0054]

Furthermore, in the case of simultaneously firing a plurality of substrate formed bodies, since an aluminum nitride sintered powder with a prescribed particle size is interposed as a spread powder between adjacent substrate formed bodies, the amount of the powder adhering to and remaining in the substrate surfaces is decreased. As a result, the roughness of the substrate surfaces is remarkably improved and the number of steps of the finishing process after sintering can be lessened.

[Brief Description of Drawings]

Fig. 1 is a cross-sectional view of a firing furnace showing the state

where a plurality of substrate formed bodies are put in a firing container according to the present invention and simultaneously fired.

Fig. 2 is a cross-sectional view of a firing furnace showing the state where a plurality of formed bodies are put in a conventional firing container and simultaneously fired.

[Explanation of Symbols]

1: firing furnace

2: hearth

3: firing container (box for firing)

3a: container main body

3b: lid

4: substrate formed body

5: BN powder (spread powder)

6: firing container

6a: container main body

6b: lid

7: sintered powder (spread powder)



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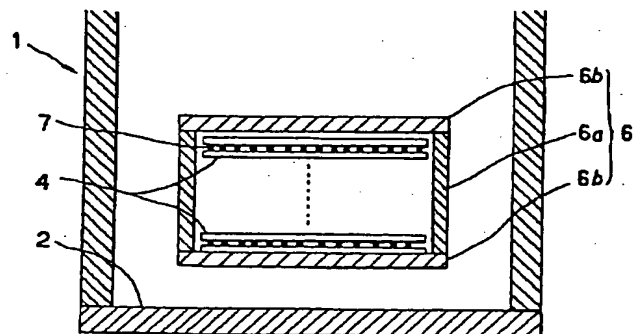
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(54) 【発明の名称】 窒化アルミニウム基板の製造方法および焼成容器

(57) 【要約】

【目的】 大型の窒化アルミニウム基板を製造する場合において焼成時に使用する焼成容器（匣）の耐久性を高めることができ、熱伝導率や表面粗度が良好な窒化アルミニウム基板を安価に量産する。

【構成】 窒化アルミニウム焼結体で形成した筒状容器本体と窒化硼素焼結体で形成され上記容器本体に被せる蓋体とから成る焼成容器内に、窒化アルミニウムを主成分とする基板成形体を收容した状態で非酸化性雰囲気中で焼結することを特徴とする。



## 【特許請求の範囲】

【請求項1】 窒化アルミニウム焼結体で形成した筒状容器本体と窒化硼素焼結体で形成され上記容器本体に被せる蓋体とから成る焼成容器内に、窒化アルミニウムを主成分とする基板成形体を収容した状態で非酸化性雰囲気中で焼結することを特徴とする窒化アルミニウム基板の製造方法。

【請求項2】 焼成容器内に複数の基板成形体を収容し、隣接する基板成形体間に窒化アルミニウムの焼結粉末をしき粉として介在させて各基板成形体を積層配置することを特徴とする請求項1記載の窒化アルミニウム基板の製造方法。

【請求項3】 しき粉としての焼結粉末の平均粒径を50～200 $\mu$ mの範囲に設定したことを特徴とする請求項2記載の窒化アルミニウム基板の製造方法。

【請求項4】 窒化アルミニウム焼結体で形成した筒状容器本体と窒化硼素焼結体で形成され上記容器本体に被せる蓋体とから成ることを特徴とする焼成容器。

## 【発明の詳細な説明】

## 【0001】

【産業上の利用分野】本発明は窒化アルミニウム基板の製造方法およびその製造方法において使用する焼成容器に係り、特に大型の窒化アルミニウム基板を製造する場合において、焼成時に使用する焼成容器（匣）の耐久性を高めることができ、熱伝導率や表面粗度が良好な窒化アルミニウム基板を安価に量産することができる窒化アルミニウム基板の製造方法および焼成容器に関する。

## 【0002】

【従来の技術】従来の金属材料と比較して強度、耐熱性、耐食性、耐磨耗性、軽量性などの諸特性に優れたセラミックス焼結体が、半導体、電子機器材料、エンジン用部材、高速切削工具用材料、ノズル、ベアリングなど、従来の金属材料の及ばない苛酷な温度、応力、摩耗条件下で使用される機械部品、構造物や装飾品材料として広く利用されている。

【0003】特に窒化アルミニウム（AlN）焼結体はAl、O、焼結体の約20倍に及ぶ高熱伝導性を有する絶縁体であり、また半導体素子を構成するシリコン（Si）に近い熱膨張係数を有することから高集積化した半導体装置の放熱板や基板、大型チップ、高出力モジュール構成材として、その用途を拡大している。

【0004】従来上記窒化アルミニウム基板は一般的に下記の製造方法によって量産されている。すなわち、窒化アルミニウム粉末にY、O、CaO等の焼結助剤と、有機バインダと、必要に応じて各種添加剤や溶媒、分散剤とを添加して原料混合体を調製し、得られた原料混合体を、例えばドクターブレード法によってシート状の成形体としたり、原料混合体をプレス成形法によって厚板状ないし大型の成形体を形成する。また複雑形状を有する製品を製造する場合には、スリップキャスト法

（泥漿鑄込法）によって成形体が調製される。

【0005】ところで、セラミックス基板の標準サイズは、一般に2インチ角（2"×2"）が主流であり、用途によっては3"×3"および4"×4"が使用される一方、特に大型用として6"×6"以上の大型基板も使用されつつある。これらの大型基板を製造するための基板成形体の成形操作は、サイズに関係なく容易であり、ドクターブレード法またはプレス成形法でも可能である。

【0006】そして得られた成形体は、空気または窒素ガス雰囲気において加熱され脱脂処理され、有機バインダとして使用された炭素、水素成分等が成形体から排除脱脂される。そして脱脂された成形体は窒素ガス雰囲気等で高温に加熱され緻密化焼結されて窒化アルミニウム基板が形成される。

【0007】上記焼結操作は、一般に図2に示すように、黒鉛（グラファイト）等の炉材を貼設した焼成炉1の炉床2上に箱状の焼成容器（焼成用匣）3を配置し、この焼成容器3内に1個または複数の脱脂した基板成形体4を収容した状態で1600～2000℃の高温に加熱して実施される。

【0008】上記基板成形体4を収容保持する焼成容器3や炉床2は、高温焼結時に成形体と反応して焼結体の特性を低下させることを防止するために、成形体と同一材料である窒化アルミニウム（AlN）焼結体や窒化硼素（BN）焼結体で形成される。上記焼成容器3は、円筒状または角筒状の容器本体3aと、容器本体3aの上端開口および下端開口それぞれ封止するために被せられる蓋体3b、3bとから成る。また複数の基板成形体4を焼成容器3内に積層配置して同時に焼成する場合には、隣接する基板成形体4、4の間にそれぞれ粒径1 $\mu$ m程度のBN粉末5をしき粉（離型剤）として介在させている。

【0009】上記のように基板成形体4と反応しない焼成容器中に基板成形体4を収容して焼結しているため、各基板成形体4の全体に熱が均一に作用し、むらの少ない均質な焼結体が得られる。また焼成炉1の炉材に含まれる炭素等の不純物が、焼成時に直接的に成形体4に作用することがなく、不純物によって焼結体に色むらや変形が発生したり、焼結体の熱伝導性が低下することが防止される。また複数の基板成形体4、4間にしき粉としてのBN粉末5を介在させているため隣接する成形体が融着するおそれが少なくなり、表面粗度が良好な窒化アルミニウム基板が得られる。

【0010】一方、上記焼成容器3を使用せず、基板成形体4を直接焼成炉1内に配置して焼成した場合には、焼成炉1の炉材から放出された過剰量の炭素蒸気によって焼結体表面が著しく損傷され、かつ焼結体全体の変形量も大きくなり、製造歩留りが急減してしまう。

【0011】上記製造方法において、原料AlN粉末と

して平均粒径が $0.3\mu\text{m}$ 以下程度の超微細な原料粉末を使用する場合は、AlN粉末単独でもかなりの緻密な焼結体が得られる。しかしながら、原料粉末表面等に付着した多量の酸素等の不純物が焼結時に、AlN結晶格子中に固溶したり、格子振動の伝播を妨げるAl-O-N化合物等の複合酸化物を生成する結果、焼結助剤を使用しないAlN焼結体の熱伝導率は低かった。

【0012】一方原料粉末として平均粒径 $0.5\mu\text{m}$ 以上のAlN粉末を使用する場合は、その原料粉末単独では焼結性が良好でないため、ホットプレス法以外には助剤無添加では緻密な焼結体を得ることが困難であり、量産性が低い欠点があった。そこで常圧焼結法によって効率的に焼結体を製造しようとする場合には、焼結体の緻密化およびAlN原料粉末中の不純物酸素がAlN結晶粒子内へ固溶することを防止するために、焼結助剤として、酸化イットリウム( $\text{Y}_2\text{O}_3$ )などの希土類酸化物や酸化カルシウムなどのアルカリ土類金属酸化物等を添加することが一般に行なわれている。

【0013】これらの焼結助剤は、AlN原料粉末に含まれる不純物酸素と反応して液相を形成し、焼結体の緻密化を達成するとともに、この不純物酸素を粒界相として固定し、高熱伝導率化も達成するものと考えられている。

【0014】

【発明が解決しようとする課題】しかしながら、上記従来の製造方法に従って特に大型のAlN基板を製造する場合においては、使用する焼成容器も大型化し、この焼成容器の耐久性の低下に起因してAlN基板の製造コストが上昇する問題点があった。すなわちBN焼結体で形成した焼成容器は、AlN製の焼成容器と比較して高温強度に優れ、また反応による劣化も少ない利点を有する一方、原料価格が非常に高い欠点を有し、さらに構造強度が低く取扱時に割れや欠けを発生し易く、基板の製造コストを押し上げる欠点があった。

【0015】一方、AlN焼結体で形成した焼成容器はBNを材料とする容器と比較して安価であり、通常の取扱い時における構造強度も十分に高い利点を有する一方で、高温では強度劣化が顕著になり、また通常の昇温速度を採用した焼結パターンでは、破壊を生じ易くなり、繰返しの使用が困難となる問題点があった。

【0016】このようにAlN焼結体およびBN焼結体で形成した焼成容器は、上記のようにいずれも一長一短を有しており、特に大型化に伴って熱衝撃によって破損し易くなる欠点があり、使用範囲や条件が狭い範囲に限定されてしまう欠点があった。

【0017】また従来の製造方法によって複数のAlN成形体を同時に焼成する際に、隣接する成形体間に粒径 $1\mu\text{m}$ の微細なBN粉末を介在させて成形体相互の融着を防止していたが、焼結完了後にAlN基板表面に付着残留するBN粉末が多くなり、表面粗度が大きくなる問

題点もあった。そのため所定の平滑面を得るためにAlN基板表面をホーニングや研磨加工によって仕上げる必要があり、AlN基板の製造工程が煩雑化する問題点もあった。

【0018】本発明は、上記問題点を解決するためになされたものであり、特に大型の窒化アルミニウム基板を製造する場合において焼成時に使用する焼成容器(匣)の耐久性を高めることができ、熱伝導率や表面粗度が良好な窒化アルミニウム基板を安価に量産することができる窒化アルミニウム基板の製造方法および焼成容器を提供することを目的とする。

【0019】

【課題を解決するための手段】本発明者らは上記目的を達成するため、焼成容器の材質および耐久性と焼結パターンとの関係、およびしき粉の種類および粒径がAlN基板の熱伝導率や表面粗度に及ぼす影響について、実験を繰り返して調査した。

【0020】その結果、容器本体および蓋体を全てBN焼結体で形成した焼成容器は、殆どの焼結パターンにおいて優れた熱衝撃耐性を有する一方、AlN焼結体製の蓋体は熱衝撃耐性が低く、通常より50%程度低い昇温速度に設定しない限り、容易に破壊してしまうことが判明した。しかしながら、AlN焼結体製の容器本体は通常の昇温速度で繰り返して使用した場合においても、優れた熱衝撃耐性を有することが確認された。

【0021】一方、製品となるAlN基板の代表利用特性である熱伝導率に及ぼす影響を確認したところ、部分的ではあっても、AlN製焼成容器を使用して焼結したAlN基板の熱伝導率は、BNで全体を構成した焼成容器で焼結したものより10~20%と著しく改善されることが判明した。

【0022】また複数の基板成形体を同時に焼成する場合において、窒化アルミニウム粉末を造粒して焼結した所定粒径の焼結粉末を隣接する基板成形体間にしき粉として介在させて焼結した場合に、隣接する基板成形体の融着が解消されるとともに、焼結後に基板表面に付着残留する粉末量が、BN粉末を使用した場合と比較して大幅に減少し、表面粗度が良好なAlN基板が得られるという知見を得た。

【0023】本発明は上記知見に基づいて完成されたものである。すなわち本発明に係る窒化アルミニウム基板の製造方法は、窒化アルミニウム焼結体で形成した筒状容器本体と窒化硼素焼結体で形成され上記容器本体に被せる蓋体とから成る焼成容器内に、窒化アルミニウムを主成分とする基板成形体を収容した状態で非酸化性雰囲気中で焼結することを特徴とする。

【0024】また、焼成容器内に複数の基板成形体を収容し、隣接する基板成形体間に窒化アルミニウムの焼結粉末をしき粉として介在させて各基板成形体を積層配置するとよい。

【0025】さらに、しき粉としての焼結粉末の平均粒径を50~200 $\mu$ mの範囲に設定するとよい。

【0026】とする請求項2記載の窒化アルミニウム基板の製造方法。

【0027】本発明方法において使用され、基板成形体の主成分となる窒化アルミニウム(AlN)粉末としては、焼結性および熱伝導性を考慮して不純物酸素含有量が3重量%以下に抑制され平均粒径が0.05~5 $\mu$ m程度、好ましくは3 $\mu$ m以下のものを使用する。

【0028】焼結助剤としては希土類元素(Y, Sc, Ce, Dyなど)の酸化物、窒化物、アルカリ土類金属(Ca)の酸化物、もしくは焼結操作によりこれらの化合物となる物質が使用され、特に酸化イットリウム(Y<sub>2</sub>O<sub>3</sub>)や酸化カルシウム(CaO)が好ましい。焼結助剤の添加量は0.5~10重量%の範囲で調整される。添加量が0.5重量%未満の場合は、焼結性の改善効果が十分に発揮されず、焼結体が緻密化されず低強度の焼結体が形成されたり、AlN結晶中に酸素が固溶し、高い熱伝導率を有する焼結体が形成できない。一方添加量が10wt.%を超える過量となると、粒界相が焼結体中に残存したり、熱処理により除去される粒界相の体積が大きいため、焼結体中に空孔が残ったりして収縮率が増大し、変形を生じ易くなる。

【0029】焼結時に使用する焼成容器の構成材料としては、AlN成形体と反応しない窒化アルミニウム(AlN)粉末および窒化硼素(BN)粉末の少なくとも一方から調製される。特に筒状容器本体と、この本体に被せる平板状の蓋体とから成る焼成容器において、焼成時に大きな熱応力が作用する蓋体を、熱衝撃特性が優れたBN焼結体で形成する一方、構造的に高い強度を有する筒状容器本体を安価なAlN焼結体で形成することにより、焼成容器全体としての耐久性を損うことなく、焼成容器の製造コストを大幅に低減することができる。

【0030】上記焼成容器としては、成形体に与える影響を可及的に抑制するために、前記の通り成形体と反応せず、また不純物含有量が少ない高純度の材料で形成することが望ましく、例えば下記の製法によって製造される。すなわち、窒化アルミニウム粉末と有機バインダとを混合した原料粉末混合体をプレス成形、スリップキャスト成形、射出成形などの種々の成形方法で処理して容器素体を形成し、その容器素体を脱脂後、窒素ガス等の還元雰囲気において温度1800~2000℃で数10時間加熱焼結処理して製造される。また、BN容器は市販されていることから、種々の形状例えば $\phi$ 400程度の大型の容器も作製できる。

【0031】一方窒化アルミニウム基板を構成する製品原料混合体の成形法としては、汎用の金型プレス法、静水圧プレス法、あるいはドクターブレード法のようなシート成形法などが適用できる。そして成形操作に引き続いて、基板成形体を非酸化性雰囲気中、例えば窒素ガス

雰囲気中で温度400~500℃に加熱して、予め添加していた有機バインダを充分に除去する。

【0032】次に脱脂処理された基板成形体は、図1に示すように窒化アルミニウム焼結体製の容器本体6aと、その容器本体6aの上下面に被せられた窒化硼素焼結体製の蓋体6b、6bとから成る焼成容器6内にAlNの焼結体粉末7を介して多段に積層される。

【0033】ここで上記焼結体粉末7は、隣接する基板成形体4が焼結時の高熱で融着することを防止するしき粉(離型剤)として作用するものである。本発明方法で使用されるしき粉としては平均粒径を50~200 $\mu$ mの範囲に設定した粗大なAlN焼結粉末を使用する。平均粒径が50 $\mu$ m未満と微細な場合には基板成形体4間に雰囲気ガスが十分に流通せず、また脱ガスも阻害されるため基板焼結体の組成が不均一になり易く、基板表面に付着残留する焼結粉末量が増大し、表面の仕上げ加工に多大な工数が必要となる。一方、平均粒径が200 $\mu$ mを超える粗大な場合にも基板表面にむらが生じ易くなる。上記焼結粉末は、市販の窒化アルミニウム原料粉末の造粒粉を脱脂焼結して製造される。

【0034】そして前記焼成容器内に収容した基板成形体の焼結操作は、窒素ガスなどの非酸化性雰囲気中で基板成形体4を含む焼成容器6全体を温度1600~2000℃に2~10時間程度加熱して実施される。焼結雰囲気は、窒素ガス、または窒素ガスを含む還元性雰囲気で行なうとよい。還元性ガスとしてはH<sub>2</sub>ガス、COガスを使用してもよい。なお、焼結は真空(僅かな還元雰囲気を含む)、減圧、加圧および常圧を含む雰囲気で行なってもよい。焼結温度が1600℃未満と低温状態で焼成すると、原料粉末の粒径、含有酸素量によって異なるが、緻密な焼結体が得にくい。

【0035】

【作用】上記構成に係る窒化アルミニウム基板の製造方法および焼成容器によれば、焼成時に大きな熱応力が作用する焼成容器の蓋体を、熱衝撃特性が優れたBN焼結体で形成する一方、構造的に高い強度を有する筒状容器本体を安価なAlN焼結体で形成しているため、焼成容器全体としての耐久性を損うことなく、焼成容器の製造コストを大幅に低減することができ、ひいてはAlN基板の製造コストを低減することができる。

【0036】また、焼成容器を部分的にAlN焼結体で形成することにより、焼結時に焼成容器内にAl<sub>2</sub>O<sub>3</sub>を還元し易い雰囲気が形成され、成形体に含有されるAl<sub>2</sub>O<sub>3</sub>量が少なくなると基板焼結体の熱伝導率が上昇する。

【0037】さらに複数の基板成形体を同時焼成する場合において、隣接する基板成形体間に所定の粒径を有する窒化アルミニウム焼結粉末をしき粉として介在させているため、従来の微細なBN粉末をしき粉として使用した場合と比較して基板表面に付着残留する粉体量が少な

くなる。その結果、基板表面の粗度が大幅に改善され、焼結後の表面仕上げ加工工数を低減することができる。

#### 【0038】

【実施例】次に下記の実施例を参照して本発明に係る窒化アルミニウム基板の製造方法および焼成容器による効果をより具体的に説明する。

#### 【0039】実施例1～5

不純物として酸素を1.0重量%含有し、平均粒径1.5 $\mu$ mの窒化アルミニウム粉末に対して、焼結助剤としてのY、O、(酸化イットリウム)を3重量%添加し、さらに溶媒および有機バインダを添加して24hr湿式混合してスラリーを調製した。次にこのスラリーをドクターブレード法によって成形し、厚さ0.8mmのシート状成形体を形成し、さらにシート状成形体を打抜きプレスによって打ち抜くことにより、1辺が200mmの正方形形状の基板成形体を多数調製し、引き続き各成形体を空气中で温度375℃で2時間加熱して脱脂処理した。

【0040】一方、平均粒径が1.5 $\mu$ mのAlN粉末にバインダーを5%添加してスプレードライ法により造粒し、この原料混合体をプレス成形して焼成容器素体を形成した。この焼成容器素体としては、内径300mm、軸方向長さ(高さ)30mm、肉厚6mmの容器本体6aと、この本体に被せる直径312mm、肉厚6mmの蓋体6bとを作成した。そして各焼成容器素体を400℃で2時間加熱して脱脂処理した後に、AlN製容器中に収容してN<sub>2</sub>雰囲気中で1900℃で6時間焼成した。そして実施例1～5および比較例1～3の焼結体を形成するための焼成容器3、6を調製した。また、3a、3bは、それぞれ6a、6bと同形状のものを購入した。

【0041】一方、平均粒径1.5 $\mu$ mの高純度AlN粉末を造粒して40～200 $\mu$ mに顆粒化して、窒素雰囲気中において1900℃で4時間焼成し、冷却後、通篩することにより、しき粉としてAlN焼結粉末を調製した。

【0042】次に前記工程で脱脂処理した基板成形体を、図1に示すように8枚ずつまとめて焼成容器6内に多段に積層配置し、この焼成容器6をN<sub>2</sub>ガスを封入した焼成炉内に収容した。各焼結回分処理毎に表1左欄に

示す平均粒径を有するしき粉を隣接する基板成形体間に介在させた。そして焼成炉1内の温度を1850℃まで高めた状態で4時間保持し、緻密化焼結を実施し、それぞれ6"×6"、厚さ0.65mmである実施例1～5に係るAlN基板焼結体を8枚ずつ調製した。

#### 【0043】比較例1

一方、図2に示すように、容器本体3aを含めて全てBN焼結体から成る焼成容器3を使用した点以外は実施例1と同一条件で焼結処理して同一寸法を有する比較例1に係るAlN基板焼結体を調製した。

#### 【0044】比較例2

また、図2に示すように容器本体3aを含めて全てBN焼結体から成る焼成容器3を使用した以外は実施例2と同一条件で焼結処理して同一寸法を有する比較例2に係るAlN基板焼結体を調製した。

#### 【0045】比較例3

さらに容器本体および蓋ともに全てAlN焼結体で形成した焼成容器を使用した以外は実施例2と同一条件で処理して同一寸法の比較例3に係るAlN基板焼結体を調製した。

【0046】そして得られた実施例1～5および比較例1～3に係る各AlN基板焼結体の特性を評価するため、その熱伝導率を測定するとともに、各基板表面の中心線平均粗さRa(JIS B0601)を測定し、下記表1右欄に示す結果を得た。

【0047】またAlN基板の製造コストに大きな影響を及ぼす焼成容器の耐久性および製造コストを算出し、表1に併記した。なお各焼成容器の耐久性は、繰返しの焼成操作において容器本体や蓋体に割れや破損を生じるまでの繰返しの使用可能回数を求め、比較例1に示すところの全てBN焼結体で形成した焼成容器の使用可能回数を基準値100として相対値で示した。また焼成容器の製造コストは、原料購入から成形、焼結、加工に至るまでの原料費および加工費を合算し、同じく比較例1の場合を基準値100として相対値で示した。

#### 【0048】

【表1】

試料No	焼成容器材料		しき粉		AlN基板特性		焼成容器	
	容器本体	蓋体	種類	平均粒径 ( $\mu\text{m}$ )	熱伝導率 ( $\text{W}/\text{m}\cdot\text{K}$ )	表面粗さ $R_a$ ( $\mu\text{m}$ )	耐久性	製造コスト
実施例1	AlN	BN	AlN焼結粉	50	221	0.2	95	45
実施例2	AlN	BN	BN粉末	1	218	0.5	95	50
実施例3	AlN	BN	AlN焼結粉	100	225	0.2	95	45
実施例4	AlN	BN	AlN焼結粉	150	231	0.2	95	45
実施例5	AlN	BN	AlN焼結粉	200	226	0.2	95	45
比較例1	BN	BN	AlN焼結粉	50	194	0.5	100	100
比較例2	BN	BN	BN粉末	1	201	0.2	100	100
比較例3	AlN	AlN	BN粉末	1	198	0.3	25	30

【0049】表1に示す結果から明かなように、AlN焼結体製容器本体6aとBN焼結体製蓋体6bとから成る焼成容器6を使用して焼結した実施例1～5に係るAlN基板焼結体においては、BN焼結体またはAlN焼結体で全てを形成した焼成容器を使用して形成した比較例1～3のAlN基板焼結体と比較して熱伝導率が10%程度上昇しており、放熱性が高い焼結体を得られた。

【0050】また従来より粗大な粒径を有するAlN焼結粉末をしき粉として使用しているため、基板表面に付着して残留するしき粉量が大幅に低減し、基板の表面粗さも改善された。

【0051】そして、焼成時に大きな熱応力が作用する

蓋体6bを、熱衝撃特性が優れたBN焼結体で形成する一方、構造的に高い強度を有する筒状容器本体6aを安価なAlN焼結体で形成することにより、焼成容器6全体としての耐久性を損うことなく、焼成容器6の製造コストを大幅に低減することができ、AlN基板自体の製造原価も低減することができた。

【0052】

【発明の効果】以上説明の通り本発明に係る窒化アルミニウム基板の製造方法および焼成容器によれば、焼成時に大きな熱応力が作用する焼成容器の蓋体を、熱衝撃特性が優れたBN焼結体で形成する一方、構造的に高い強度を有する筒状容器本体を安価なAlN焼結体で形成しているため、焼成容器全体としての耐久性を損うことな

く、焼成容器の製造コストを大幅に低減することができる。

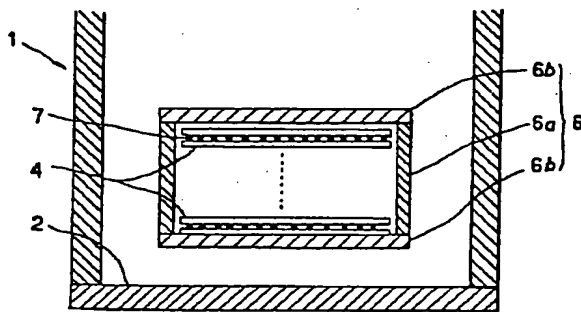
【0053】また、焼成容器を部分的にAlN焼結体で形成することにより、焼結時に焼成容器内にAl、O<sub>2</sub>を還元し易い雰囲気形成され、成形体に含有されるAl、O<sub>2</sub>量が少なくなって基板焼結体の熱伝導率が上昇する。

【0054】さらに複数の基板成形体を同時焼成する場合において、隣接する基板成形体間に所定の粒径を有する窒化アルミニウム焼結粉末をしき粉として介在させているため、BN粉末をしき粉として使用した場合と比較して基板表面に付着残留する粉体量が少なくなる。その結果、基板表面の粗度が大幅に改善され、焼結後の表面仕上げ加工工数を低減することができる。

【図面の簡単な説明】

【図1】本発明に係る焼成容器内に複数の基板成形体を

【図1】



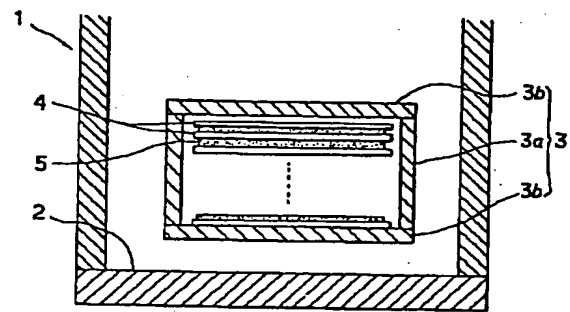
収容し、同時に焼成する状態を示す焼成炉の断面図。

【図2】従来の焼成容器内に複数の成形体を収容し同時に焼成する状態を示す焼成炉の断面図。

【符号の説明】

- 1 焼成炉
- 2 炉床
- 3 焼成容器（焼成用匣）
- 3a 容器本体
- 3b 蓋体
- 4 基板成形体
- 5 BN粉末（しき粉）
- 6 焼成容器
- 6a 容器本体
- 6b 蓋体
- 7 焼結粉末（しき粉）

【図2】



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